

U.S. Application No.
Unassigned

International Application No.
PCT/BE99/00025

Attorney Docket No.
VANM172.001APC

Date: August 24, 2000

Page 1

**TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 USC 371**

International Application No.: PCT/BE99/00025
International Filing Date: 18 February 1999
Priority Date Claimed: 26 February 1998
Title of Invention: GRANULATED BREAD IMPROVER FOR THE PREPARATION OF
BAKERY PRODUCTS
Applicant(s) for DO/EO/US: Filip ARNAUT et al.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 USC 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 USC 371.
3. ☒ This express request to begin national examination procedures (35 USC 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 USC 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 USC 371(c)(2))
 - a) ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b) ☒ has been transmitted by the International Bureau.
 - c) ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 USC 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 USC 371(c)(3))
 - a) ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b) ☐ have been transmitted by the International Bureau.
 - c) ☒ have not been made; however, the time limit for making such amendments has NOT expired.
 - d) ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 USC 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 USC 371(c)(4)).
10. ☒ A copy of the International Preliminary Examination Report with any annexes thereto, such as any amendments made under PCT Article 34.
11. ☐ A translation of the annexes, such as any amendments made under PCT Article 34, to the International Preliminary Examination Report under PCT Article 36 (35 USC 371(c)(5)).

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Items 11. to 16. below concern other document(s) or information included:

12. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
13. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
14. ☒ A FIRST preliminary amendment.
☐ A SECOND or SUBSEQUENT preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A power of attorney and/or address letter.
17. ☒ International Application as published. (COVER SHEET ONLY)
18. ☐ Small Entity Statement.
19. ☒ PCT Form PCT/IPEA/402.
20. ☒ PCT Form PCT/IB/308.
21. ☐ PCT request form.
22. ☒ A return prepaid postcard.
23. ☒ The following fees are submitted:

| | | | | FEES |
|--|-----------------|-----------------|-------|-------|
| BASIC FEE | | | | \$840 |
| CLAIMS | NUMBER FILED | NUMBER EXTRA | RATE | |
| Total Claims | 13 - 20 = | 0 × | \$18 | \$0 |
| Independent Claims | 2 - 3 = | 0 × | \$78 | \$0 |
| Multiple dependent claims(s) (if applicable) | | | \$260 | \$0 |
| TOTAL OF ABOVE CALCULATIONS | | | | \$840 |
| Reduction by 1/2 for filing by small entity (if applicable). Verified Small Entity statement must also be filed. (NOTE 37 CFR 1.9, 1.27, 1.28) | | | | \$ |
| TOTAL NATIONAL FEE | | | | \$840 |
| TOTAL FEES ENCLOSED | | | | \$840 |
| amount to be refunded: | | | | \$ |
| amount to be charged: | | | | \$ |

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24. (X) A check in the amount of \$840 to cover the above fees is enclosed.
25. (X) Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40 per property.
26. (X) The Commissioner is hereby authorized to charge only those additional fees which may be required, now or in the future, to avoid abandonment of the application, or credit any overpayment to Deposit Account No. 11-1410. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

KNOBBE, MARTENS, OLSON & BEAR, LLP
620 Newport Center Drive
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Newport Beach, CA 92660



Signature

Thomas R. Arno
Printed Name

40,490
Registration Number

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VANM172.001APC

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Arnaut et al.) Group Art Unit Unknown
Appl. No. : Unknown)
Filed : Herewith)
For : GRANULATED BREAD)
IMPROVER FOR THE)
PREPARATION OF BAKERY)
PRODUCTS)
Examiner : Unknown)

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, D.C. 20231

Dear Sir:

Prior to examination of the above-captioned patent application, please amend the application as follows:

IN THE SPECIFICATION:

Please amend the specification as follows.

At page 1, line 19, delete "and state of the art";
page 4, line 21, replace "Aims" with --Summary--;
page 5, line 1, delete "General description of the invention";
page 6, before line 24, please insert --Brief Description of the Drawings

The figures of 1 to 4 represent the average size distribution
of various improvers according to the invention compared
to the powder formulation of the starting material.--; and

page 8, please delete lines 14-18.

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IN THE CLAIMS:

Please cancel existing Claims 1-12.

Please add the following new claims:

13. A bread improver in the form of a powder, comprising:
agglomerated particles of fat and enzymes having a mean particle size of at least 250 μm .
14. The improver of Claim 13, wherein the particles further comprise proteins.
15. The improver of Claim 13, wherein the mean particle size is between about 300 and 2000 μm .
16. The improver of Claim 13, wherein the ratio of the standard deviation/mean agglomerated particle size is less than 0.8.
17. The improver of Claim 13, wherein the particles further comprise ingredients selected from the group consisting of: emulsifiers, sugars, organic acids, minerals, and a combination thereof.
18. The improver of Claim 13, further comprising a carrier.
19. The improver of Claim 18, wherein the carrier is selected from the group consisting of starch, wheat flour and soy flour.
20. A method for producing a bread improver, comprising:
preparing a starting material in the form of a powder, comprising particles of fat and enzymes having a mean particle size less than 200 μm ;
spraying the powder with an atomized liquid; and
recovering the agglomerated bread improver.
21. The method of Claim 20, wherein the ratio of the standard deviation/mean agglomerated particle size of the agglomeration is lower than the ratio of the standard deviation/mean particle size of the starting material.
22. The method of Claim 20, wherein the liquid is water.
23. The method of Claim 20, wherein the liquid is an agglomerating agent selected from the group consisting of polysaccharides, proteins, and a mixture thereof.
24. The method of Claim 20, wherein the fluidizing is performed in a fluidized bed reactor.

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25. The method of Claim 24, wherein the temperature of the fluidized bed reactor is between about 20°C and 45°C.

REMARKS

The foregoing amendments are made to correct minor informalities and more particularly describe Applicant's invention. No new matter has been added.

Should the Examiner have any questions with respect to this document or the application in general, he is invited to contact the undersigned attorney.

Please charge any additional fees, including any fees for additional extension of time, or credit overpayment to Deposit Account No. 11-1410. A duplicate copy of this sheet is enclosed.

Respectfully submitted,

KNOBBE, MARTENS, OLSON & BEAR, LLP

Dated: 8/24/00

By: 

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5

GRANULATED BREAD IMPROVER FOR THE PREPARATION OF BAKERY
PRODUCTS

10

Field of the invention

The present invention is related to a granulated bread improver for the preparation of bakery products, and to the bakery products comprising said improver.

The present invention is also related to the preparation process of said granulated bread improver.

Background of the invention and state of the art

Baked bread products are made of the basic ingredients: flour (mostly wheat or rye flour), water, salt and yeast. Other types of flour (barley, oats, soy, sunflower, cassava and other). The process of baking is subject to a lot of variations due to external (temperature, humidity, handling) and internal (variations of flour and yeast quality) factors.

To obtain a more reliable production process and a constant end product the baker uses ingredients and additives. These products are mostly added as one coformulated product and are known as improvers. These improvers may contain emulsifiers and fats, enzymes, sugars, organic acids, minerals, polysaccharides and

proteins. This includes products such as diacetyltartaric acid esters of monoglycerides, lactic acid ester of monoglycerides, monoglycerides, amylases, hemicellulases and pentosanases, oxidases, lipases, proteinases, glucose, fructose, sucrose, ascorbic acid, lactic acid, phosphates, sulphates, guar, locust bean gum, gluten, soy proteins.

Said list is not exhaustive and is presented as an illustration of a possible improver. A mixture of ingredients active in the preparation process of baked goods is called an improver as soon as it contains more than one class of active components mixed together as to optimise by a synergetic effect their performances in the baking process. The mixing of several enzymes does not lead to obtain an improver. On the other hand, mixing one or more enzymes with an emulsifier (for example DATEM) or with ascorbic acid gives the formulation of an improver.

The improver can be formulated as a liquid, as a paste or more commonly as a powder. The powdered formulation contains the active ingredients as above-mentioned and a carrier substance. The carrier substance is added to the active ingredients in order to dilute them to the suitable concentration. The improver will be added by the baker in a concentration of 0.5 to 20% to the flour.

The most currently used carrier substances are starch, wheat flour or soy flour. The powdered formulation of the improver is very stable, and easy to weigh and to store.

However, said formulation presents also various drawbacks. If one of the components is an emulsifier or another fat-based product, the improver will tend to stick, which may result in more or less compact lumps.

This effect is more pronounced at higher temperatures. As a result of its poor free flowing characteristics, the improver is not easily carried by automatic dosing systems and tends to get stuck in the
5 pipes. This also makes dosing inaccurate, and makes the cleaning of the pipes arduous. In addition, a flour which contains a lot of small particles may give rise to allergic reactions for sensitised persons, for instance as in baker's disease which is well known to be caused by
10 allergenic wheat flour particles and by the powdered additives.

In various fields, it has been proposed to increase the particle size of various compositions.

The enzyme producers have granulated their
15 enzymes to reduce this risk of allergenicity as illustrated in the US Patent US-4,940,665.

The International Patent Application WO93/07260 describes the manufacturing of dust free particles of enzymes by spray-coating solid particulate
20 cores.

The European Patent Application EP-A-0289069 describes also oil or fat obtained in a granular form.

The European Patent Application EP-A-0659344 describes a dry yeast composition consisting of dry
25 inactive yeast and a bread improving agent which is preferably in a granular form or consists of small particles adhering larger yeast granules. This European Patent Application also describes a process to obtain a mixture of hemicellulase, amylase and Vitamin C in a
30 granular form.

One might try to overcome the disadvantages of a powder form improver by applying the teaching of the

European Patent Application EP-A-0659344 and thus producing or buying granulated ingredients or mixtures of ingredients. This however will result in a non-homogeneous particle distribution. In the specific applications
5 described in the European Patent Application EP-A-0659344, said drawbacks are not important, because the end product is packed under reduced pressure afterwards, becomes a firm block and the particles are not able to move relative to one other. Mixing all improver ingredients in one solution
10 and granulating as described in the European Patent Application EP-A-0659344 will only be efficient if all ingredients are miscible and stable in water. This is mostly not the case for bread improvers. This is illustrated by the fact that common emulsifiers as
15 diacetyltartaric ester of monoglycerides or stearylactylates are readily hydrolysed in water and can not be used in aqueous solutions. It is also impossible to make aqueous solutions incorporating enzymes when at least one protease is to be present in the mixture.

20

Aims of the invention

The main aim of the invention is to provide a bread improver in the form of a powder and its preparation process which do not present the drawbacks of the above-
25 mentioned state of the art.

The main aim of the invention is to provide a bread improver in the form of a powder and its preparation process, which presents improved free-flowing properties, which is more easy to handle and which may reduce the risk
30 of potential allergenicity.

General description of the invention

The present invention is related to a bread improver in the form of a powder which is made of agglomerated particles having a mean particle size of at least 250 μm . The mean agglomerated particle size of this product is preferably comprised between 300 and 2000 μm .

Preferably, the standard deviation /mean agglomerated particle size ratio is lower than 0.8, preferably lower than 0.65 .

10 In a preferred embodiment of the present invention, the particles are made of at least 2 different active ingredients, advantageously balanced to obtain an effect in bakery products.

The improver according to the invention can further comprise one or more ingredients selected from the group consisting of emulsifiers, fats, enzymes, sugars, organic acids, minerals, polysaccharides, proteins and/or a mixture thereof.

A second aspect of the present invention is related to a method for obtaining the granulated improver in the form of a powder according to the invention, said method comprising the steps of :

- preparing a starting material being a bread improver in the form of a dried powder having a mean particle size lower than 200 μm ,
- introducing and maintaining said starting material in a fluidised bed reactor, under spraying of an atomised liquid, in order to obtain an agglomeration of the dried powder particles of said material, and
- 30 - recovering a bread improver in the form of a dried powder made of agglomerated particles having mean particle size of at least 250 μm .

In the method according to the invention, the standard deviation/mean agglomerated particle size ratio of the agglomerated particles is preferably lower than the standard deviation/mean agglomerated particle size ratio of
5 said starting material.

In the method according to the invention, said liquid comprises water and preferably further comprises an agglomerating agent selected from the group consisting of polysaccharides (such as guar, alginate,
10 carrageenan, pectin, maltodextrins) or proteins (such as gelatin) and/or a mixture thereof.

In the method according to the invention, the temperature of the fluidised bed reactor is preferably comprised between 20 and 45 °C, more preferably between 25
15 and 40 °C.

Advantageously, the fluidised bed reactor used in the method according to the invention is a Glatt granulating device. However, other fluidised bed equipments are also suitable for the preparation of the bread improver
20 according to the invention. Examples are Allgaier Wirbelschichttrockner (Allgaier Uchingen, GERMANY) and Vector Fluid Bed (Vector Marion, Iowa, USA).

Detailed description of the invention

25 The granulation of a formulated improver is not obvious to perform. Due to its very complex composition, special care has to be taken to keep the activity of the different components intact. Specially sensitive active components are enzymes and emulsifiers.
30 The temperature sensitivity of enzymes is commonly known. Emulsifiers are also temperature sensitive : they melt at a

certain temperature, but even before this temperature is reached, an emulsifier tends to become sticky and will cause unwanted lumping in the improver. This means that the improver temperature during granulation must be kept under
5 this melting temperature.

Moreover, emulsifiers are sensitive to moisture.

The dry improver may also contain other ingredients that are unstable in water or that tend to
10 react with other ingredients present in the improver. As an illustration one can mention the reaction between bromate and cysteine. Therefore the moisture content must be kept low during the entire granulation process.

The granulation step is followed by measuring
15 the particle size distribution by using the Coulter LS 200 Particle Analyser.

The bread improver powder is introduced in the granulating device. Warm air, preferably dried, is blown through the powder as to fluidise it. The air flow is
20 such that a stable fluidised bed is obtained. This result is measured and determined by a person skilled in the art according to bed expansion and movement of the particles in said fluidised bed. The temperature of the fluidised bed has to be lower than 45 °C. Preferably it is lower than 40
25 °C. The lower limit is defined by the evaporative capacity of the air and is usually higher than 25 °C. Once the fluidised bed is stabilised, water or preferably an aqueous solution containing an agglomerating material is sprayed on the fluidised bed. This step is usually called "topspray".
30 It is also possible to granulate using the "bottom spray" method (also called the "Wurster process"). The aqueous

solution, which preferably contains an agglomerating agent or a mixture of agglomerating agents dissolved in water, is sprayed on the bread improver powder. Examples of these agglomerating agents are: polysaccharides (such as guar, alginate, carrageenan, pectin, maltodextrins) or proteins (such as gelatin). The bread improver powder (possibly additionally dried) having the suitable mean particle size is then recovered from the granulating device.

10 The invention will be further illustrated in the following examples, in view of the enclosed figures, without limiting the scope of the present invention.

Short description of the drawings

15 The figures 1 to 4 represent the average size distribution of various improvers according to the invention compared to the powder formulation of the starting material.

20 Examples

Example 1 : Small scale granulation

The granulation was performed using a Glatt GPCG 1 (Glatt-Binzen-Germany).

25 The starting material is made of 1250g of the product S500 Controller, available in Belgium from Puratos N.V. and is put in the granulation vessel. Some of the ingredient of S500 product are DATEM, enzymes, ascorbic acid ,sugar and dry wheat flour.

30 A solution of 0.66% guar in water was used as the spraying solution at a spray rate of about 7.6 ml/min. The atomisation pressure in the nozzle was about 0.5 bar. Air inlet temperature was about 51 °C and the linear air

speed about 3.5 m/s. Bed temperature was maintained at about 29 °C. It fluctuated between about 26 °C and 32 °C. Process time was about 75 minutes. In this period 3.8 g of guar was sprayed on the product.

5 In the figure 1, it is shown that the average particle size distribution is considerably increased by the granulation process. In all figures, (1) indicates the particle size distribution prior to the treatment, while (2) shows the particle size distribution after the
10 agglomeration treatment according to the invention.

Example 2 : Baking experiments

Baking tests of Belgian pistolets were performed using the following method. The ingredients used
15 in the four experiments as specified in Table 1 are all expressed in grams :

Table 1 : Ingredients for baking tests 1 to 4

| Ingredients | Test 1 | Test 2 | Test 3 | Test 4 |
|-------------------------------------|--------|--------|--------|--------|
| Wheat flour | 1500 | 1500 | 1500 | 1500 |
| Water | 930 | 930 | 930 | 930 |
| Yeast | 90 | 90 | 90 | 90 |
| Salt | 30 | 30 | 30 | 30 |
| Non-granulated S500 | 30 | | 45 | |
| S500 after granulation experiment 1 | | 30 | | 45 |

20 After mixing to optimum dough development (Diosna spiral mixer), the dough was allowed to rest for a total time of 25 minutes, then the dough was divided in 66 g dough pieces (Eberhardt divider) and allowed to rise

for 15 minutes during which the dough pieces are cut. Final proofing takes 70 minutes and then thepistolets are baked at 230 °C for 20 minutes in a oven with steam (Miwe Condo).

After baking the specific volume is measured
5 based on the rapeseed displacement method. The standard deviation on the baking experiment is about 0.2 litre/kg.

Table 2 : Results for the example 2

| | Test 1 | Test 2 | Test 3 | Test 4 |
|------------------------------|--------|--------|--------|--------|
| Specific volume (l/kg flour) | 12.9 | 12.8 | 14.1 | 14.0 |

10 Example 3 : Small scale granulation

The granulation was performed using a Glatt GPCG 1 (Glatt-Binzen-Germany).

The starting material is made of 1250 g of the product Joker, available in Belgium and in Italy from
15 Puratos N.V. and is put in the granulation vessel. Some of the ingredients of Joker product are: enzymes, ascorbic acid and dry wheat flour. It contains no emulsifier or fat. A solution of 25% maltodextrines (Maldex150-Amylum Belgium) in water was used as the spraying solution at a spray rate
20 of about 4.4 ml/min. The atomisation pressure in the nozzle was about 1.5 bar. Air inlet temperature was about 51 °C and the linear air speed about 3.5 m/s. Bed temperature was maintained at about 29 °C. It fluctuated between 26 °C and 32 °C. Process time was about 150 minutes. In this period
25 165 g of maltodextrines was sprayed on the product.

In the figure 2, it is shown that the average particle size distribution is considerably increased by the granulation process according to the invention.

Example 4 : Industrial granulation of improver

The industrial granulator was a WSG 120-200 from Glatt - Binzen(Germany) equipped with 3 nozzles. The granulated product has the following composition :

| | | |
|---|---------------------------|------------|
| 5 | dextrose | : 50 kg |
| | DATEM in powder form | : 25 kg |
| | dry wheat flour | : 117.5 kg |
| | pre-diluted hemicellulase | : 3.75 kg |
| | pre-diluted alpha-amylase | : 3.76 kg |

10 The enzymes were prediluted in wheat flour to facilitate a reproducible mixing. The ingredients were mixed and introduced in the granulation bowl. A solution of 0.66% guar in water was used as the spraying solution at a spray rate of about 25 l/h. The atomisation pressure in
15 the nozzles was about 2 bar. Air inlet temperature varied between 20 and 75 °C. The inlet air flow was about 3800 m³/h. Bed temperature was set at about 30 °C. It fluctuated between 27 °C and 35 °C. Process time was about 80 minutes. In this period, 0.5 kg of guar was sprayed on the product.

20 Figure 3 and 4 and Table 3 show that starting from a particle size distribution with at least 3 different populations, the product obtained after 80 minutes presents a uniform distribution, with an increased mean particle size.

25

Summary of results

Results are expressed before and after granulation in the Table 3.

30

Table 3 : **Results for examples 1, 3 and 4**

| | Example 1 | | Example 3 | | Example 4 | |
|------------------------------|-----------------|----------------|-----------------|----------------|-----------------|----------------|
| | Before gran. | After gran. | Before gran. | After gran. | Before gran. | After gran. |
| Median (μm) | 151 | 454 | 79 | 608 | 88 | 851 |
| Mean (μm) | 238 | 531 | 92 | 617 | 100 | 890 |
| < 250 μm (vol. %) | 74 | 17 | 98 | 4 | 97 | 13 |
| < 50 μm (vol. %) | 3.9 | 0.5 | 30 | 0.4 | 31 | 0.7 |
| Standard deviation (SD) | 245 | 326 | 64 | 200 | 72 | 466 |
| SD/mean | 1.03 | 0.61 | 0.70 | 0.32 | 0.72 | 0.52 |

The value of SD/mean is a measure for the
 5 uniformity of distribution. A more uniform particle size
 distribution has industrial advantages. In the table, it is
 clear that the granulation step decreases the value of
 SD/mean, which results in a more uniform distribution
 (steeper, more distinct peaks).

10 It is clear that the exact granulation
 parameters must be adapted to the exact composition and
 especially to the carrier substance. If gums or other
 agglomerating substances are already present in the dry
 improver mix, it can be sufficient to spray only water to
 15 granulate the powder as such.

CLAIMS

1. Bread improver in the form of a powder, characterised in that it is made of agglomerated particles having a mean particle size of at least 250 μm .
- 5 2. Improver according to claim 1, characterised in that the mean agglomerated particle size is comprised between 300 and 2000 μm .
3. Improver according to claim 1 or 2, characterised in that the standard deviation/mean
10 agglomerated particle size ratio is lower than 0.8, preferably lower than 0.65 .
4. Improver according to any of the claims 1 to 3, characterised in that the particles are made of at least fat and proteins.
- 15 5. Improver according to claim 4, characterised in that the particles further comprise ingredients selected from the group consisting of emulsifiers, enzymes, sugars, organic acids, minerals, polysaccharides and/or a mixture thereof.
- 20 6. Improver according to the claim 4 or 5, characterised in that the particles further comprise a carrier, preferably selected from the group consisting of starch, wheat flour soy flour.
7. Method for obtaining the granulated bread
25 improver according to any of the preceding claims, said method comprising the steps of :
 - preparing a starting material being a bread improver in the form of a dried powder having a mean particle size lower than 200 μm ,
 - 30 - introducing and maintaining said starting material in a fluidised bed reactor, under spraying of an atomised liquid, in order to obtain an agglomeration of the dried

powder particles of said material, and

- recovering a bread improver in the form of a dried powder made of agglomerated particles having mean particle size of at least 250 μm .

5 8. Method according to claim 7, characterised in that the standard deviation/mean agglomerated particle size ratio of the agglomerated particles is lower than the standard deviation/mean agglomerated particle size ratio of said starting material.

10 9. Method according to claim 7 or 8, characterised in that the liquid comprises water.

 10. Method according to claim 9, characterised in that the liquid further comprises an agglomerating agent selected from the group consisting of
15 polysaccharides (such as guar, alginate, carrageenan, pectin, maltodextrins) or proteins (such as gelatin) and/or a mixture thereof.

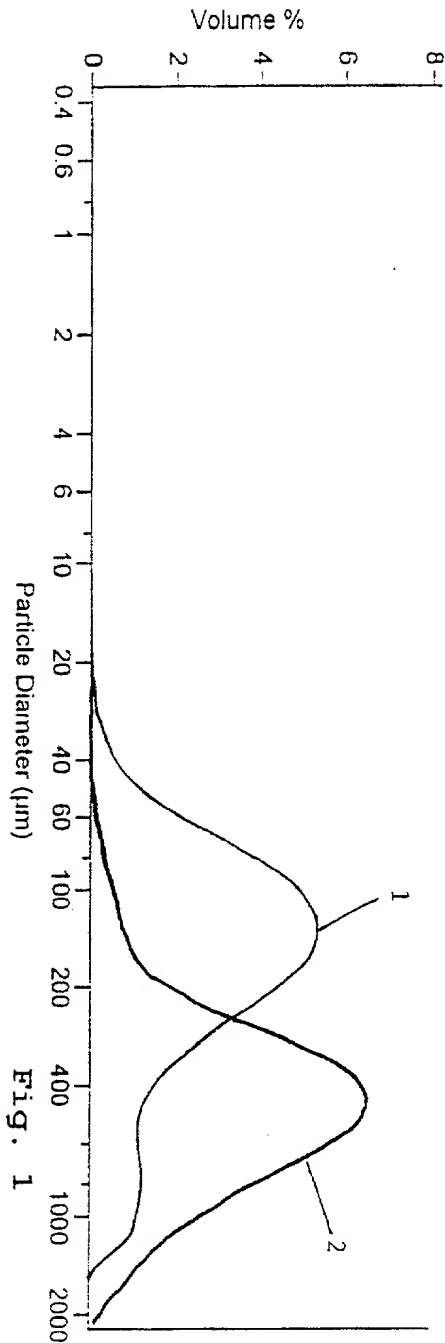
 11. Method according to any of the claims 7 to 10, characterised in that the temperature of the
20 fluidised bed reactor is comprised between 20 and 45 $^{\circ}\text{C}$, more preferably between 25 and 40 $^{\circ}\text{C}$.

 12. Method according to any of the claims 7 to 11, characterised in that the fluidised bed reactor is a Glatt granulating device.

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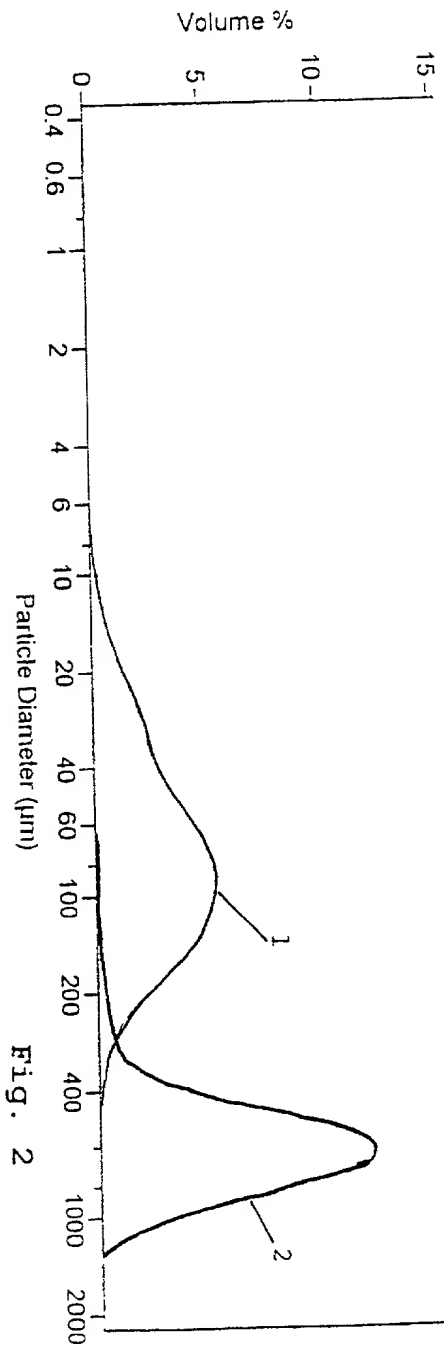


Fig. 2

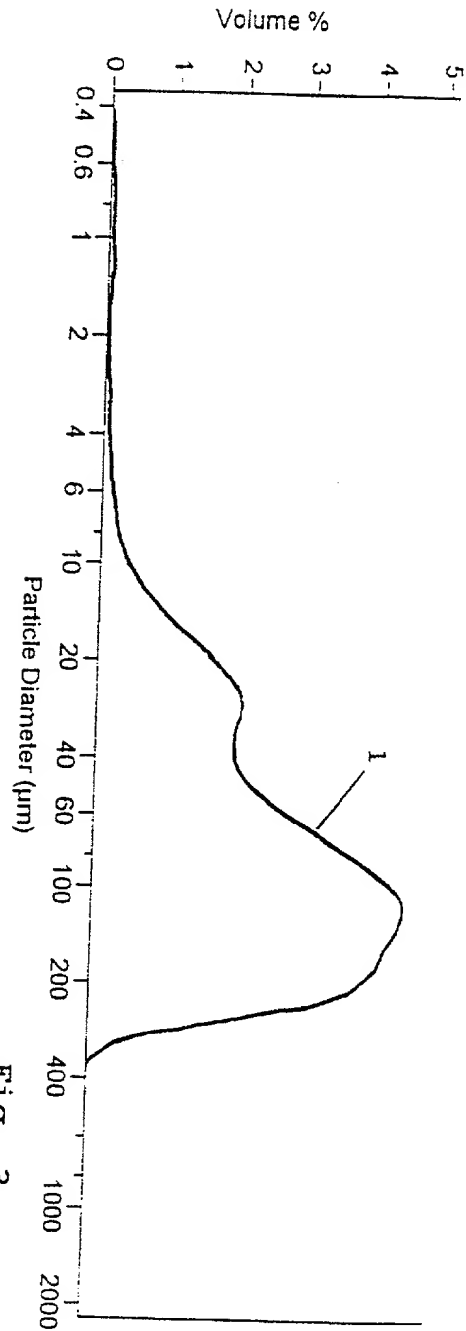


Fig. 3

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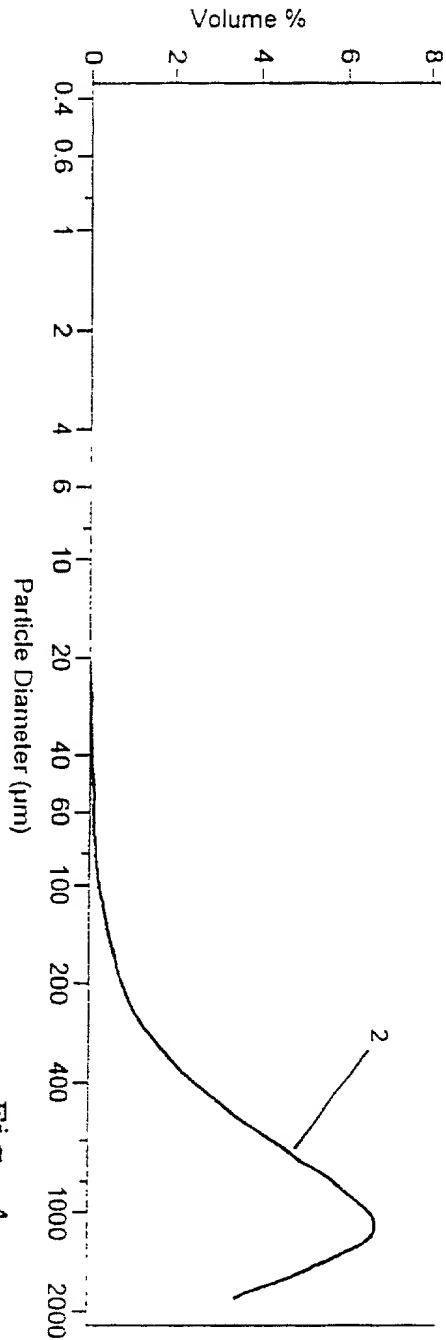


Fig. 4

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Declaration and Power of Attorney for Patent Application

Déclaration et Pouvoirs pour demandes de brevet

French Language Declaration

En tant que l'inventeur nommé ci-après, je déclare par le présent acte que :

Mon domicile, mon adresse postale et ma nationalité figurant ci-dessous à côté de mon nom,

Je crois être le premier inventeur original et unique (si un seul nom est mentionné ci-dessous), ou l'un des premiers co-inventeurs originaux (si plusieurs noms sont mentionnés ci-dessous) du sujet revendiqué, pour lequel une demande de brevet a été déposée concernant l'invention intitulée :

et dont les caractéristiques sont fournies ci-joint à moins que la case suivante n'ait été cochée :

- ☐ a été déposé le
sous le numéro de Demande des Etats-Unis ou
sous le numéro de demande internationale
PCT et modifiée le
(le cas échéant).

Je déclare par le présent acte avoir passé en revue et pris connaissance du contenu des caractéristiques ci-dessus, revendications comprises, telles que modifiées par tout amendement dont il aura été fait référence ci-dessus.

Je reconnais de voir divulguer toute information pertinente à l'examen de cette demande, comme le définit le Titre 37, §1.56 du Code fédéral des réglementations.

As a below named inventor, I hereby declare that :

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled :

the specification of which is attached hereto unless the following box is checked :

- ☒ was filed on 18 February 1999
as United States Application Number or PCT
International Application Number
PCT/BE99/00025 and was amended on
14 Feb. 2000 (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56.

French Language Declaration

Je revendique par le présent acte avoir la priorité étrangère, en vertu du Titre 35, § 119 du Code des Etats-Unis, sur toute demande étrangère de brevet ou certificat d'inventeur figurant ci-dessous et ai aussi pris connaissance de toute demande étrangère de brevet ou de tout certificat d'inventeur ayant une date de dépôt précédant celle de la demande à propos de laquelle une priorité est revendiquée.

Prior foreign applications

Demande(s) de brevet antérieure(s)

(Number) (Country)
(Numéro) (Pays)

(Number) (Country)
(Numéro) (Pays)

(Number) (Country)
(Numéro) (Pays)

Je revendique par le présent acte tout bénéfice, en vertu du Titre 35, § 120 du Code des Etats-Unis, de toute demande de brevet effectuée aux Etats-Unis figurant ci-dessous et, dans la mesure où le sujet de chacune des revendications de cette demande de brevet n'est pas divulgué dans la demande américaine préalable, en vertu des dispositions de premier paragraphe du Titre 35, § 112 du Code des Etats-Unis, je reconnais devoir divulguer toute information pertinente à la demande de brevet comme défini dans le Titre 37, § 1.56 du Code fédéral des réglementations, dont j'ai pu disposer entre la date de dépôt de la première demande et la date de dépôt de la demande nationale ou PCT internationale :

(Application Serial No.) (Filing date)
(No. de série de la demande) (Date de dépôt)

(Application Serial No.) (Filing date)
(No. de série de la demande) (Date de dépôt)

Je déclare par le présent acte que toute déclaration ci-incluse est, à ma connaissance, véridique et que toute déclaration formulée à partir de renseignements ou de suppositions est tenue pour véridique; et de plus, que toutes ces déclarations ont été formulées en sachant que toute fausse déclaration volontaire ou son équivalent est passible d'une amende ou d'une incarcération, ou des deux, en vertu de la Section 1001 du Titre 18 du Code des Etats-Unis et que de telles déclarations volontairement fausses risquent de compromettre la validité de la demande de brevet ou du brevet délivré à partir de celle-ci.

I hereby claim foreign priority under Title 35, United States Code, § 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed.

Priority claimed
Droit de priorité revendiqué

| | | |
|----------------------------|----------------------------------|-----------------------|
| 26/02/1998 | <input checked="" type="radio"/> | <input type="radio"/> |
| (Day/Month/Year Filed) | Yes | No |
| (Jour/Mois/Année de dépôt) | Oui | Non |
| | <input type="radio"/> | <input type="radio"/> |
| (Day/Month/Year Filed) | Yes | No |
| (Jour/Mois/Année de dépôt) | Oui | Non |
| | <input type="radio"/> | <input type="radio"/> |
| (Day/Month/Year Filed) | Yes | No |
| (Jour/Mois/Année de dépôt) | Oui | Non |

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application :

(Statut) (Status)
(Breveté, en attente, annulé) (Patented, pending, abandoned)

(Statut) (Status)
(Breveté, en attente, annulé) (Patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful and false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application of any patent issued thereon.

French Language Declaration

POUVOIRS : En tant que l'inventeur cité, je désigne par la présente l'(les) avocat(s) et/ou agent(s) suivant(s) pour qu'il(s) poursuive(nt) la procédure de cette demande de brevet et traite(nt) toute affaire avec le Bureau des brevets et marques s'y rapportant.

(mentionner le nom et le numéro d'enregistrement)

POWER OF ATTORNEY : As named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and trademark Office connected there with.

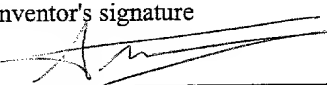
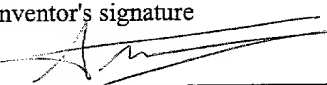
(list name and registration number)

Adresser toute correspondance à :

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(nom et numéro de téléphone)

Direct Telephone Calls to :
(name and telephone number)

| | |
|--|--|
| Nom complet de l'unique ou premier inventeur <u>1-00</u> | Full name of sole or first inventor <u>ARNAUT, Filip</u> |
| Signature de l'inventeur  | Inventor's signature  |
| Date | Date <u>7/7/00</u> |
| Domicile | Residence <u>Weverstraat 58 - B-1761 ROOSDAAL (BELGIUM)</u> BEX |
| Nationalité | Citizenship <u>BELGIAN</u> |
| Adresse postale | Post Office Address <u>Weverstraat 58 - B-1761 ROOSDAAL (BELGIUM)</u> |

(Fournir les mêmes renseignements et la signature de tout co-inventeur supplémentaire)

(Supply similar information and signature for any subsequent joint inventor)

| | |
|---|---|
| Nom complet du second co-inventeur, le cas échéant <i>2-00</i> | Full name of second joint inventor, if any JANKE, Hans-Christian |
| Signature du second inventeur <i>H.C. Janke</i> | Second inventor's signature <i>H.C. Janke</i> |
| Date <i>19.07.2000</i> | Date <i>19.07.2000</i> |
| Domicile | Residence Timmerbruch 31 - D-57368 LENNESTADT (GERMANY) <i>DE</i> |
| Nationalité | Citizenship GERMAN |
| Adresse postale | Post Office Address Timmerbruch 31 - D-57368 LENNESTADT (GERMANY) |

| | |
|---|---|
| Nom complet du troisième co-inventeur, le cas échéant | Full name of third joint inventor, if any |
| Signature du troisième inventeur | Third inventor's signature |
| Date | Date |
| Domicile | Residence |
| Nationalité | Citizenship |
| Adresse postale | Post Office Address |

| | |
|---|--|
| Nom complet du quatrième co-inventeur, le cas échéant | Full name of fourth joint inventor, if any |
| Signature du quatrième inventeur | Fourth inventor's signature |
| Date | Date |
| Domicile | Residence |
| Nationalité | Citizenship |
| Adresse postale | Post Office Address |